Measuring Fluoride in Water and Wastewater using the Thermo Scientific Orion Dual Star pH/ISE Meter

Water and Lab Products, Thermo Fisher Scientific

Key Words
Thermo Scientific Orion Dual Star pH/ISE meter, fluoride ion selective electrode (ISE), fluoride, wastewater, water

Goal
This technical note explains how to accurately measure fluoride content in general water and wastewater samples using the Thermo Scientific™ Orion™ Dual Star™ pH/ISE dual channel benchtop meter.

Introduction
Too much or too little fluoride can be harmful to humans and the environment. Precise measurement of fluoride by municipalities, potable water plants and bottled water manufacturers promotes quality water for consumption and helps prevent damage to the environment.

Required Equipment
- Orion Dual Star pH/ISE meter with electrode stand (Cat. No. 2115000)
- Fluoride combination electrode (Cat. No. 9609BNWP)
- Automatic Temperature Compensation (ATC) probe (Cat. No. 927007MD)
- Stirrer probe (Cat. No. 096019) or magnetic stir plate and bar
- 1 ppm fluoride standard premixed with Total Ionic Strength Adjustment Buffer (TISAB) II (Cat. No. 040906)
- 2 ppm fluoride standard premixed with TISAB II (Cat. No. 040907)
- TISAB II solution (Cat. No. 940909)
- Thermo Scientific™ Orion™ Optimum Results™ A electrode filling solution (Cat. No. 900061)
- Deionized water
- 100 mL volumetric flasks
- 50 mL beakers
- 50 mL, 25 mL and 15 mL pipettes
- 50 mL graduated cylinders or 10 mL automatic pipette
Fluoride Electrode Setup

Note: Add filling solution each day before using the electrode. The filling solution level should be at least one inch above the level of sample to ensure a proper flow rate. The fill hole should always be open when taking measurements.

See Figure 1 for labeled electrode components.

1. Remove the protective shipping cap from the sensing element and save the cap for storage.

2. Insert the spout of the Optimum Results A bottle into the electrode fill hole, add a small amount of filling solution to the chamber, tip the electrode upside-down to wet the O-ring and return the electrode to its original, vertical position.

3. Hold the electrode body and use your thumb to push down on the electrode cap to allow a few drops of filling solution to drain out and wet the junction.

4. Release the electrode cap and allow the outer sleeve to return to its original position. If the outer sleeve does not return to its original position immediately, check if the O-ring is moist and repeat step 3.

5. Add filling solution to the electrode up to the bottom of the electrode fill hole.

6. Soak the electrode in the 1 ppm fluoride calibration standard until all of the equipment is ready for use.

Calibration Standard Preparation

1. Prepare a 0.5 ppm fluoride standard by pipetting 50 mL of the 1 ppm fluoride standard premixed with TISAB II (Cat. No. 040906) and 25 mL of the TISAB II solution (Cat. No. 940909) into a 100 mL volumetric flask. Dilute to the mark with deionized water. Mix the solution well.

2. Using a graduated cylinder or automatic pipette, add 30 mL of the 0.5 ppm fluoride standard to a 50 mL beaker and label the beaker.

3. Using a graduated cylinder or automatic pipette, add 30 mL each of the 1 ppm fluoride standard and 2 ppm fluoride standard to separate 50 mL beakers and label the beakers.
Sample Preparation

1. Using a graduated cylinder or pipette, measure and add 15 mL of the sample to a 50 mL beaker and label the beaker. Add 15 mL of TISAB II to the beaker. Gently stir the sample and TISAB II to mix the solution.

2. Repeat step 1 for additional samples.

Meter Preparation

The Orion Dual Star meter has two BNC, reference and ATC connections. These connections are labeled as Channel 1 or Channel 2 on the ridge above the connections.

1. Prepare the power adapter by selecting the appropriate wall outlet plug and sliding the plug plate into the groove on the back of the adapter.

2. Connect the power adapter to the meter and then to the wall outlet. See Figure 2. Connect the fluoride electrode to one of the BNC inputs on the meter and note which channel (channel 1 or channel 2) was selected. See Figure 3. Connect the ATC probe to the 8 pin MiniDIN input on the meter and note which channel was selected. See Figure 4. Connect the stirrer probe to the stir jack input on the meter. See Figure 5.

Note: It is highly recommended that the EZ Startup menu be completed the first time that the meter is used. The EZ Startup menu sets important meter parameters, such as the displayed language, date and time, measurement mode and read type for each channel, and data output settings. To access the EZ Startup menu from the measurement mode, press the setup key, press the ▲/▼ keys to highlight EZ Startup and press the f2 (select) key.

1. In the measurement mode, press the setup key.

2. Press the ▲/▼ keys to highlight Channel 1 or Channel 2, depending on which BNC input the fluoride electrode was connected to, and press the f2 (select) key.

3. Press the ▲/▼ keys to highlight Measure Mode and press the f2 (select) key. Press the ▲/▼ keys to highlight ISE and press the f2 (accept) key. Press the ▲/▼ keys to highlight ppm (or mg/L, depending on your preference or method) and press the f2 (accept) key.

4. Press the ▲/▼ keys to highlight Electrode ID and press the f2 (select) key. Press the ▲/▼ keys to highlight F- and press the f2 (accept) key.

5. Press the ▲/▼ keys to highlight Resolution and press the f2 (select) key. Press the ▲/▼ keys to highlight 3 Significant Figures (1.00) and press the f2 (accept) key.

6. Press the ▲/▼ keys to highlight Temperature Input and press the f2 (select) key. Press the ▲/▼ keys to highlight ATC1 or ATC2, depending on which 8 pin MiniDIN input the ATC probe was connected to, and press the f2 (accept) key.

7. Press the ▲/▼ keys to highlight Calibration Setup and press the f2 (select) key. Press the ▲/▼ keys to highlight Autoblank and press the f2 (select) key. Press the ▲/▼ keys to highlight On and press the f2 (accept) key. Verify that the Low Level Stability and Isopotential options are set to Off. Press the f1 (back) key.

8. Press the ▲/▼ keys to highlight Read Type and press the f2 (select) key. Press the ▲/▼ keys to highlight On Ready and press the f2 (select) key.

9. Press the f1 (back) key.

10. Press the ▲/▼ keys to highlight Instrument Parameters and press the f2 (select) key.

11. Press the ▲/▼ keys to highlight Export Data (PC/Printer/Log) and press the f2 (select) key. Press the ▲/▼ keys to highlight Export Trigger and press the f2 (select) key. Press the ▲/▼ keys to highlight Channel 1 or Channel 2, depending on which BNC input the fluoride electrode was connected to, and press the f2 (accept) key. Press the ▲/▼ keys to highlight Data Log and press the f2 (select) key. Press the ▲/▼ keys to highlight Data Log On/Off and press the f2 (select) key. Press the ▲/▼ keys to highlight On and press the f2 (accept) key.
12. Press the \texttt{f1 (back)} key twice.
13. Press the \texttt{\textasciitilde / \textasciitilde} keys to highlight \texttt{Stirrer Speed} and press the \texttt{f2 (select)} key. Press the \texttt{\textasciitilde / \textasciitilde} keys to highlight 1 and press the \texttt{f2 (accept)} key. Press the \texttt{f1 (back)} key.
14. Press the \texttt{f1 (back)} key. The meter will return to the measurement mode.

**Calibration Procedure**

1. Prepare and condition the fluoride electrode. Connect the fluoride electrode, ATC probe and stirrer probe to the meter (note which channel the fluoride electrode is connected to) and place the electrode and probes in the electrode stand. Prepare the 2 ppm, 1 ppm and 0.5 ppm standards.

2. In the measurement mode, press the \texttt{cal} key.

3. Dual channel display only: Press the \texttt{\textasciitilde / \textasciitilde} keys to highlight Channel 1 or Channel 2 as the channel to calibrate and press the \texttt{f2 (accept)} key.

4. Rinse the fluoride electrode, ATC probe and stirrer probe with deionized water, blot dry and place into the 0.5 ppm calibration standard.

5. When the electrode and standard are ready, press the \texttt{f3 (start)} key to begin the calibration.

6. Wait for the concentration value to stop flashing and then use the numeric keypad and the \texttt{decimal} key to enter the concentration of the first standard as 0.50 and press the \texttt{f2 (accept)} key.

7. Press the \texttt{f2 (next)} key to proceed to the next calibration standard.
8. Rinse the fluoride electrode, ATC probe and stirrer probe with deionized water, blot dry and place into the 1 ppm calibration standard.

9. When the electrode and standard are ready, press the \texttt{f3 (start)} key.

10. Wait for the concentration value to stop flashing and then use the numeric keypad and the \texttt{decimal} key to enter the concentration of the second standard as 1.00 and press the \texttt{f2 (accept)} key.

11. Press the \texttt{f2 (next)} key to proceed to the next calibration standard.

12. Rinse the electrode, ATC probe and stirrer probe with deionized water, blot dry and place into the 2 ppm calibration standard.

13. When the electrode and standard are ready, press the \texttt{f3 (start)} key.

14. Wait for the concentration value to stop flashing and then use the numeric keypad and the \texttt{decimal} key to enter the concentration of the third standard as 2.00 and press the \texttt{f2 (accept)} key.
15. Press the f3 (cal done) key. A summary of the
calibration will be displayed. The slope should be -54
mV per decade to -65 mV per decade.

16. Press the f2 (log/print) key to save and end the
calibration and export the calibration data to the
calibration log.

Sample Measurement Procedure
1. Calibrate the fluoride electrode and meter (note
which channel the fluoride electrode is connected to)
and make sure that the electrode and probes are in
the electrode stand. Prepare the fluoride samples.

2. Rinse the fluoride electrode, ATC probe and stirrer
probe with deionized water, blot dry and place into
the sample. Press the stirrer key to turn on the stirrer
probe.

3. The meter display will flash stabilizing and then show
ready once the measurement is stable. Record the
concentration and temperature of the sample when
the meter display shows ready. When ready is shown
on the display, the meter will export the measurement
to the data log.

4. Press the stirrer key to turn off the stirrer probe.
Remove the fluoride electrode, ATC probe and stirrer
probe from the sample.

5. Repeat steps 2 through 4 for all of the samples.

6. When all of the samples have been measured, store
the equipment. For short term storage between
samples and overnight, store the fluoride electrode in
the 1 ppm fluoride calibration standard. Store the
ATC probe and stirrer probe dry.
Visit www.thermoscientific.com/water to view the full line of Thermo Scientific Orion products and download product literature, user guides and manuals and software updates. For any questions or technical support, contact your local Technical Sales Representative or our Technical Support Specialists by phone at 1-978-232-6000 or 1-800-225-1480 (US toll-free), or by email at wai.techservbev@thermofisher.com.

**Ordering Information**

To purchase an Orion Dual Star meter, electrodes and other related products, please contact your local equipment distributor and reference the part numbers listed below.

<table>
<thead>
<tr>
<th>Product</th>
<th>Description</th>
<th>Part Number</th>
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<tr>
<td>Meter</td>
<td>Orion Dual Star pH/ISE Dual Channel Benchtop Meter</td>
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<tr>
<td>Electrodes</td>
<td>Orion Fluoride Combination Electrode</td>
<td>96098NWP</td>
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<td>Orion Stainless Steel ATC Temperature Probe</td>
<td>927007MD</td>
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<tr>
<td>Solutions</td>
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<td>Orion 2 ppm Fluoride Standard Premixed with TISAB II, 475 mL</td>
<td>040907</td>
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<td></td>
<td>Orion 10 ppm Fluoride Standard Premixed with TISAB II, 475 mL</td>
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<td>Orion TISAB II Solution, 3.8 L (1 gallon)</td>
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<td></td>
<td>Orion Optimum Results A Electrode Filling Solution, 5 x 60 mL</td>
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<td>Swing Arm Electrode Stand</td>
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